

DOCKET NO.: 5926P037

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re the Application of:

PAUL FREDERICK FEWSTER

Application No.:

Filed:

For: **X-Ray Diffraction Apparatus and  
Method**

Art Group:

Examiner:

Commissioner for Patents  
P.O. Box 1450  
Alexandria, VA 22313-1450

**REQUEST FOR PRIORITY**

Sir:

Applicant respectfully requests a convention priority for the above-captioned application, namely:

COUNTRY	APPLICATION NUMBER	DATE OF FILING
Great Britain	GB 0208572.8	13 April 2002

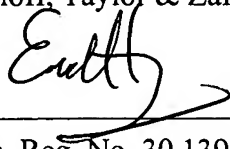
☐ A certified copy of the document is being submitted herewith.

Respectfully submitted,

Blakely, Sokoloff, Taylor & Zafman LLP

Dated: 10/12/09

12400 Wilshire Boulevard, 7th Floor  
Los Angeles, CA 90025  
Telephone: (310) 207-3800

  
Eric S. Hyman, Reg. No. 30,139



PCT/NL 0.3 / 91927-200  
Rec'd PCT/PTO 12 OCT 2003



INVESTOR IN PEOPLE

12

REC'D 19 JUN 2003

WIPO PCT

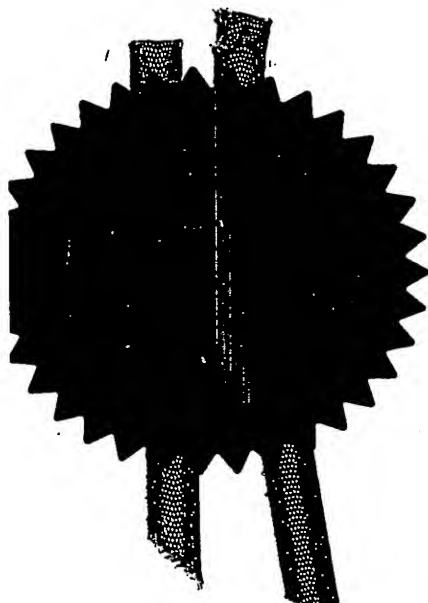
The Patent Office  
Concept House  
Cardiff Road  
Newport  
South Wales  
NP10 8QQ

I, the undersigned, being an officer duly authorised in accordance with Section 74(1) and (4) of the Deregulation & Contracting Out Act 1994, to sign and issue certificates on behalf of the Comptroller-General, hereby certify that annexed hereto is a true copy of the documents as originally filed in connection with the patent application identified therein.

In accordance with the Patents (Companies Re-registration) Rules 1982, if a company named in this certificate and any accompanying documents has re-registered under the Companies Act 1980 with the same name as that with which it was registered immediately before re-registration save for the substitution as, or inclusion as, the last part of the name of the words "public limited company" or their equivalents in Welsh, references to the name of the company in this certificate and any accompanying documents shall be treated as references to the name with which it is so re-registered.

In accordance with the rules, the words "public limited company" may be replaced by p.l.c., plc, P.L.C. or PLC.

Re-registration under the Companies Act does not constitute a new legal entity but merely subjects the company to certain additional company law rules.



*G. Hany*

Signed

Dated 9 May 2003

**PRIORITY  
DOCUMENT**

SUBMITTED OR TRANSMITTED IN  
COMPLIANCE WITH RULE 17.1(a) OR (b)

The  
Patent  
Office15APR02 E710995-1 DC 008  
P01/7700 0.00-0208572.8

1/77

0208572-8

THE PATENT OFFICE  
A

13 APR 2002

NEWPORT

The Patent Office  
Cardiff Road  
Newport  
Gwent NP10 8QQRequest for grant of a patent  
See notes on the back of this form. You can  
also get an explanatory leaflet from the Patent  
Office to help you fill in this form)

Your reference

PHGB020040

Patent application number  
(The Patent Office will fill in this part)

0208572.8

13 APR 2002

Full name, address and postcode of the or of  
each applicant (underline all surnames)KONINKLIJKE PHILIPS ELECTRONICS N.V.  
GROENEWOUDSEWEG 1  
5621 BA EINDHOVEN  
THE NETHERLANDS

Patents ADP Number (if you know it)

7419294001

If the applicant is a corporate body, give the  
country/state of its incorporation

THE NETHERLANDS

Title of the invention

X-RAY DIFFRACTION APPARATUS AND METHOD

Name of your agent (if you have one)  
"Address for service" in the United Kingdom  
to which all correspondence should be sent  
(including the postcode)Brian T. STEVENS  
Philips Intellectual Property and Standards  
Cross Oak Lane  
Redhill  
Surrey RH1 5HA

Patents ADP number (if you know it)

8359165500

If you are declaring priority from one or more  
earlier patent applications, give the country  
and the date of filing of the or of each of these  
earlier applications and (if you know it) the or  
each application number

Country

Priority Application number  
(if you know it)Date of filing  
(day/month/year)If this application is divided or otherwise  
derived from an earlier UK application, give  
the number and the filing date of the earlier  
application

Number of earlier application

Date of filing  
(day/month/year)Is a statement of inventorship and of right to  
grant of a patent required in support of this  
request? (Answer "Yes" if:

YES

- a) any applicant named in part 3 is not an inventor, or
  - b) there is an inventor who is not named as an  
applicant, or
  - c) any named applicant is a corporate body.
- See note (d))

Enter the number of sheets for any of the following items you are filing with this form.  
Do not count copies of the same document.

Continuation sheets of this form

Description	15 ✓	
Claims(s)	3 ✓	CF
Abstract	1 ✓	
Drawings	5 <u>only</u>	

1. If you are also filing any of the following, state how many against each item:

Priority Documents

Translations of priority documents

Statement of inventorship and right

to grant of a patent (*Patents Form 7/77*)

Request for preliminary examination and

search (*Patents Form 9/77*)

Request for substantive examination

(*Patents Form 10/77*)

Any other documents

(*Please specify*)

I/We request the grant of a patent on the basis of this application.

Signature

*B.T. Turner*

Date *12-04-2002*

Name and daytime telephone number of person to contact in the United Kingdom

01293 815492

(R. Turner)

arning

er an application for a patent has been filed, the Comptroller of the Patent Office will consider whether publication or communication of the invention should be prohibited or restricted under Section 22 of the Patents Act 1977. You will be informed if it is necessary to prohibit or restrict your invention in this way. Furthermore, if you live in the United Kingdom, section 23 of the Patents Act 1977 stops you from applying for a patent abroad without first getting written permission from the Patent Office unless an application has been filed at least 6 weeks beforehand in the United Kingdom for a patent for the same invention and either no direction prohibiting publication or communication has been given, or any such direction has been revoked.

tes

If you need help to fill in this form or you have any questions, please contact the Patent Office on 0645 500505.

Write your answers in capital letters using black ink or you may type them.

If there is not enough space for all the relevant details on any part of this form, please continue on a separate sheet of paper and write "see continuation sheet" in the relevant part(s). Any continuation sheet should be attached to this form.

If you have answered "Yes" *Patents Form 7/77* will need to be filed.

Once you have filled in the form you must remember to sign and date it.

For details of the fee and ways to pay please contact the Patent Office.

## DESCRIPTION

## X-RAY DIFFRACTION APPARATUS AND METHOD

5 The invention relates to an X-ray diffraction apparatus for high resolution diffractometry and a method of high resolution X-ray diffractometry.

The analysis of high quality thin layer materials, especially near perfect  
10 single crystal thin layer materials is valuable in determining layer thickness and composition of those materials. This approach may be used, for example, in the semiconductor industry.

High resolution X-ray diffraction is a well established technique for carrying out the analysis of such materials.

15 X-ray diffractometers have been used for many years for analysing perfect, nearly perfect and highly imperfect materials on a routine basis.

An early high resolution diffractometer is the double-crystal diffractometer which is illustrated in Figure 1. An X-ray source 101 transmits X-rays to a collimating crystal 103 which directs them onto a sample 105 which  
20 can be rotated about an axis 107. The X-rays are diffracted by the sample 105 onto a detector 109. The sample is then rotated and the intensity of X-rays reaching the detector is measured as a function of the angle of this rotation. This structure is still widely used, and gives good results especially on perfect and near-perfect samples

25 Unfortunately, double crystal diffractometers have a number of disadvantages. Firstly, the Bragg angle of the collimating crystal 103 has to match that of the sample 105. The matching of the collimating crystal and the sample crystal is very important and the resolution is fixed by the crystals. Accordingly, a different collimating crystal 103 is required for each new sample  
30 material.

Secondly, the X-rays in a double crystal diffractometer reach a large and ill-defined region of the sample. Since this ill-defined region is the region

analysed, X-ray diffractometers do not give good results for non crystalline materials or materials with defects.

Thirdly, in a double crystal diffractometer a number of different wavelengths are diffracted into the detector simultaneously. This reduces the precision and resolution of the results if the sample 105 is bent.

Fourthly, there is a large background level of illumination that reaches the detector from imperfect samples which makes analysis of any results a problem.

Furthermore, the double crystal diffractometer is very sensitive to the alignment and significant changes can be observed with a tilt angle varying by only  $0.2^\circ$ . This makes the double crystal diffractometer difficult to set up.

A number of proposals to alleviate some of these problems have been made.

Bartels describes a diffractometer which removes the inconvenience of having to change the crystal in "Characterization of Thin Layers on Perfect Crystals with a Multipurpose High Resolution X-ray Diffractometer", Bartels, Journal Vacuum Science and Technology B Volume 1 page 338 (1983). Bartels replaced the collimating crystal of a double crystal diffractometer with two channel-cut monochromators which provide a good monochromator with a very narrow angular divergence. However, this change to the double-crystal diffractometer geometry does not solve many of the problems of the double-crystal diffractometer. In particular, the Bartels diffractometer cannot be used for interpreting complex diffraction patterns from all but the most perfect and flat samples.

Another prior art design is a triple-crystal diffractometer which overcomes the problem of a bent sample and produces a significantly improved diffraction profile. This is illustrated in Figure 2. The triple crystal diffractometer differs from the double crystal diffractometer by having a further analysing crystal 104 interposed between the sample 105 and the detector 109. Each of the crystals is independently rotatable. The analyser acts as a detector with a very narrow angular acceptance.

Unfortunately, away from the position in which all three crystals have the same scattering angle the recorded intensity profile recorded at the detector broadens rapidly. Furthermore, alignment is very difficult and this configuration has accordingly only been used by a few research groups.  
5 Moreover, each new sample still requires a new collimating crystal.

The current state of the art commercial instrument is a multiple crystal diffractometer, in which the collimating crystal of the triple crystal diffractometer is replaced by a multiple crystal collimating arrangement, and the analyser has a channel cut crystal rotated about the axis of the sample.

10 Prior art high resolution systems are complex and difficult to set up and in particular take a significant time to record data since it is necessary to rock the sample, i.e. to adjust the angular position, to collect data. It would be beneficial to reduce the time taken to make measurements. Of course, such an increase in speed is particularly beneficial in production environments,  
15 though also worthwhile in any application.

Commercial diffractometers may be used in particular for quality control. In practice it is the double crystal approach that has traditionally been used, and the very difficult alignment of the triple-axis diffractometer is normally avoided. Although the multiple crystal diffractometer is an excellent research  
20 instrument, it has not replaced the simpler double crystal diffractometer for production line control. In view of the disadvantages of the double crystal configuration mentioned above there is a need for an alternative X-ray diffractometer suitable for routine use that addresses these disadvantages.

25 According to a first aspect of the invention there is provided an X-ray apparatus for high-resolution X-ray diffraction, comprising: a sample stage for holding a sample having a front face with the front face oriented substantially normally to a predetermined normal direction; a means for generating a collimated beam of X-rays at a predetermined target location on the sample  
30 stage at an angle of between  $0^\circ$  and  $60^\circ$  to the normal direction, the beam having an angular divergence at the sample stage in the range  $0.01^\circ$  to  $0.20^\circ$ ; and an X-ray detector arranged laterally of the sample stage for detecting X-

rays scattered by the sample to a predetermined range of angles to the normal direction, the angles in the predetermined range being in the range from  $80^\circ$  to  $90^\circ$ .

The inventive approach accordingly makes use of a means for  
5 generating a collimated beam of X-rays, a sample stage and a position sensitive detector. To take a measurement, a sample is mounted on the stage. The configuration of these components and the choice of reflection may be arranged to require no movement, or a very small movement of the detector for large detection angles, during data collection. Nevertheless, in  
10 spite of the simplicity of the arrangement high-resolution results may be obtained as explained below.

The prior art instruments such as a double-crystal or multiple-crystal diffractometer overcome wavelength dispersion of the X-ray source by various methods. Without compensation, the wavelength dispersion would broaden  
15 the measured peaks and destroy the high resolution required. For example, a multiple-crystal diffractometer works by isolating a single required wavelength. In contrast, the apparatus according to the invention overcomes dispersion by geometric arrangement.

To measure the X-rays scattered at a range in angles to create a profile  
20 of X-ray intensity versus angle the double-crystal and multiple-crystal diffractometers rotate the sample.

It might at first be thought that it would not be possible to achieve high resolution diffractometry by measuring a range of angles simultaneously. In order to carry out diffractometry in parallel, with a range of angles entering the  
25 detector at once, it is necessary that the beam used to probe the sample has a divergent range of angles. This could easily reduce the resolution.

However, by using the arrangement of the present invention, the scanning or angular spread is achieved through geometry. This has the significant benefit that a position sensitive X-ray detector may be used to  
30 measure the X-rays diffracted by the sample at different angles at the same time, i.e. in parallel, not sequentially. Accordingly, the use of the invention can greatly speed up the process of obtaining measurements from a sample.

Hence, useable data can be collected from a typical semiconductor layer structure in one to ten seconds where a conventional high-resolution diffractometer would take one to ten minutes for comparable data.

5 The X-ray detector is located to measure X-rays scattered to a predetermined range of angles which predetermined range falls within the range of  $80^\circ$  to  $90^\circ$  inclusive from the normal direction. In other words, the X-ray detector measures X-rays diffracted by the sample that are emitted in a direction closely parallel to the surface.

10 This ensures that the angle subtended by a finite size illuminated area of the sample when viewed at the detector is as small as possible, thus increasing the resolution. As will be appreciated, the closer the diffracted beam gets to being parallel to the surface the smaller the projected area is as seen from the detector and accordingly the higher the resolution of the detector. Thus, preferably the detector is positioned to measure X-rays  
15 diffracted into the range of  $85^\circ$  to  $90^\circ$ , further preferably  $88^\circ$  to  $90^\circ$ .

A further feature of the invention is that the means for generating a collimated X-ray beam is arranged to generate a collimated beam that is not too far from the normal to the sample plane. As will be explained later, this geometry is useful in order that wavelength variation and beam spread do not  
20 give rise to excessive reduction in the high resolution. Preferably, the beam angle is less than  $40^\circ$  to the normal to the sample.

Preferably, the X-ray detector has a linear resolution in the normal direction of less than (i.e. better than) 0.002 times the distance from the X-ray detector to the predetermined target location. Further preferably, the linear  
25 resolution is better than 0.001 times the distance from the X-ray detector to the predetermined target location, or further preferably 0.0005 times the distance from the X-ray detector to the predetermined target location.

The means for generating a collimated beam of X-rays is preferably an X-ray source and a slit between the X-ray source and the sample stage. This  
30 allows a suitable beam to be provided without excessive difficulty or expense. Indeed, it is of particular benefit that the invention does not require an

expensive monochromator to select only one X-ray wavelength to be incident on the sample.

Preferably, the X-ray source has a dimension of no more than 0.2mm in the direction normal to the beam in the plane containing the normal, the incident beam and the scattered X-rays.

Preferably, the X-ray detector is an elongate X-ray detector extending in a direction parallel to the normal direction for detecting in parallel X-rays diffracted by the sample as a function of distance along the normal direction and hence over a predetermined range of angles to the normal direction. This allows measurements to be made in parallel over a range of angles. In an alternative arrangement a slit may be provided in front of an X-ray detector, and the slit moved to scan a range of angles.

Conveniently, the position sensitive X-ray detector is a solid state detector.

In another aspect, the invention relates to an X-ray apparatus for high-resolution X-ray diffraction of a sample having a substantially single crystal thin layer, comprising: a sample stage for holding a sample having a front face with the front face oriented substantially normally to a predetermined normal direction; an X-ray source for directing X-rays towards the sample stage; a slit arranged between the X-ray source and the sample stage, the X-ray source and slit being dimensioned and arranged to direct a collimated beam of X-rays at a predetermined target location on the sample stage at an angle of between  $0^\circ$  and  $60^\circ$  to the normal direction; an elongate X-ray detector arranged laterally of the sample stage for detecting X-rays diffracted by the sample as a function of distance along the length of the X-ray detector and hence over a predetermined range of angles greater than  $80^\circ$  to the normal direction.

According to a further aspect of the invention, there is provided an X-ray apparatus for high-resolution X-ray diffraction of a sample of predetermined type, comprising: a sample stage for holding a sample having a front face with the front face oriented substantially normally to a predetermined normal direction; a means for generating a collimated beam of X-rays at a predetermined target location on the sample stage at an angle of between  $0^\circ$

and  $60^\circ$  to the normal direction; and an X-ray detector arranged laterally of the sample stage for detecting X-rays diffracted by the sample at an angle of greater than  $80^\circ$  to the normal direction as a function of distance along the normal direction and hence over a predetermined range of angles to the normal direction, wherein the linear resolution of the X-ray detector in the  
5 normal direction and the means for generating a collimated beam are selected to achieve an angular resolution of at least  $0.1^\circ$  with a substantially single crystal sample of the predetermined material type.

The invention also relates to the use of an X-ray apparatus as set out  
10 above to measure a substantially single crystal thin layer sample mounted on the sample stage and oriented to diffract the collimated X-ray beam onto the position sensitive X-ray detector. The thin layer is preferably a semiconductor layer.

The invention also relates to a method of high-resolution X-ray  
15 diffraction, comprising providing an X-ray source and a slit for producing a beam of X-rays; providing a sample stage having a front face and an elongate X-ray detector located laterally of the sample stage extending longitudinally along a direction substantially normal to the front face; mounting a sample having a substantially single crystal thin layer material extending in a plane on  
20 the front face of the sample stage oriented to diffract a beam of X-rays incident from the X-ray source through the slit towards a direction substantially along the plane onto the X-ray detector; directing a beam of X-rays through the slit to the sample at an angle of  $0^\circ$  to  $60^\circ$  to the normal to the plane; and measuring with the X-ray detector the X-rays diffracted by the sample as a function of  
25 position along its length.

Preferably, the step of measuring the X-rays diffracted by the sample include recording the intensity of X-rays instant on the detector simultaneously at a number of different locations along the detector.

30 Embodiments of the invention will now be described, purely by way of example, with reference to the accompanying drawings in which:

Figure 1 shows a conventional double-crystal diffractometer;

Figure 2 shows a conventional triple-access diffractometer;

Figure 3 shows a schematic drawing of an embodiment of the invention;

Figure 4 illustrates the diffraction in reciprocal space;

Figure 5 illustrates the effect of various forms of incident beam deviation  
5 on the measured result;

Figure 6 shows results from a diffractometer according to the invention;  
and

Figure 7 shows results from a prior art high resolution diffractometer for  
comparison.

10

Referring to Figure 3, the X-ray diffraction apparatus includes an X-ray enclosure 2, which holds the various components. An X-ray source 4 is arranged to emit X-rays through slit 6 towards sample stage 8. An elongate position sensitive detector 10 extends laterally of the sample stage along the  
15 normal to the front face 12 of the sample stage.

Any suitable X-ray source 4 may be used, including for example a cathode type X-ray apparatus or a synchrotron. In the specific embodiment, the X-ray source 4 produces  $\text{CuK}\alpha$  X-rays.

20

The collimator 6 is arranged in front of the source 4 to direct a beam 11 of X-rays 11 at a target location 15 on the sample stage. The beam 11 is at angle of between  $0^\circ$  and  $60^\circ$  to the normal direction 14 to the sample stage 8.

25

In use, a sample 16 is mounted on the sample stage 8. The sample has a thin film 18 on its front face 12. Thus, the source 4 and slits 6 are arranged so that a beam of X-rays hits the thin film 18 on the front face 12 of the sample 16. The incident beam is incident at an angle  $\alpha$  to the normal direction 14:  $\alpha$  is given by  $\alpha = 90^\circ - \omega$ , where  $\omega$  is the incident angle as conventionally defined.

30

The incident beam 11 is scattered or diffracted off the thin film 18 to the detector 10. The incident beam 11 is diffracted by a scattering angle  $2\theta$  to emerge at an angle closely parallel to the plane of the thin film 18 on the front face of the sample, at an angle  $\alpha'$  to the normal. Because of the strain in the film with respect to the underlying substrate and the finite thickness of the thin

film 18 the scattered beam 20 is emitted at a range of angles  $\alpha'$  to the normal 14. The range of angles  $\alpha'$  measured may be for example  $85^\circ$  to  $90^\circ$ . The position sensitive X-ray detector 10 can measure the X-ray intensity at a plurality of positions along its length simultaneously, and output this information to a data collection and recording device 22.

It will be noted that the scattered beam 20 is not at the angle that would result from a simple scattering by a plane parallel to the thin film 18 since the angle  $\alpha$  is less than  $60^\circ$ , preferably less than  $40^\circ$ , whereas the angle  $\alpha'$  is greater than  $80^\circ$ , preferably greater than  $85^\circ$ . Thus, the X-rays measured are those scattered off the thin film 18 by crystal planes that are not parallel to the thin film 18 but inclined at angles to the thin film.

The reason for this will now be explained with reference to Figures 4 and 5 which are diagrams of the arrangement of Figure 3 scattering of thin film 18 shown in reciprocal space.

Vector  $\mathbf{K}$  40 represent the input beam. Vector  $\mathbf{K}'$  42 represents X-rays scattered at approximately  $89^\circ$  to the normal 14. Vector  $\mathbf{K}''$  44 represents X-rays scattered at a slightly different angle, here  $85^\circ$ , to the normal 14. As is known from conventional scattering theory, the magnitude of X-rays emitted in a direction  $\mathbf{K}'$  is given by sample information in k-space at a location given by  $\mathbf{K}' - \mathbf{k}$ . Thus, the intensity of X-rays scattered into the direction of vector 42 is given by the intensity in k-space at radial vector 48 which is  $\mathbf{K}' - \mathbf{k}$ , whereas the intensity of radiation scattered into the direction of vector 44 is given by the intensity in k space of radial vector 50 given by  $\mathbf{K}'' - \mathbf{k}$ . Thus, it will be seen from the drawing that a detector 10 that detects X-rays scattered to angles from the normal 14 of  $85^\circ$  to  $89^\circ$  samples information along a line 52 in k-space parallel to normal 14.

It should be noted that for samples that are substantially single crystalline the samples are accurately crystalline in the directions along the plane of the sample and accordingly give rise to a good 2-dimensional array of points in this plane. In contrast, the finite thickness of the thin film and possible inaccuracies and variations in its thickness give rise to sample information around each of the reciprocal lattice points in k-space that varies in

the direction of the normal 14. This variation in k-space gives information about the quality of the thin film. The sample information in k-space is thus represented by the points of the reciprocal crystal lattice spread substantially in the direction normal 14 to the thin film but with little spread parallel to the thin film.

The X-ray detector detects the intensity of scattered X-ray radiation along its length, i.e. along a line parallel to the normal direction 14, and in so doing effectively scans nearly parallel to the line 52 in k-space and in so doing measures information about the quality of the thin film.

The geometric arrangement has further significant benefits which will now be described with reference to Figure 5. Variation in wavelength of the incident beam causes uncertainty of the region in k-space scanned as represented diagrammatically by oval 54 in Figure 5a. However, because the distribution of scattering around each reciprocal lattice point in k-space is effectively along line 52 normal to the sample surface, the spread in wavelength and corresponding spread in the sampled region of k-space 54 does not give rise to a significant smearing of the results since the spread 54 is not close to parallel to the line 52.

To explain in more detail, Figure 5a illustrates a nominal k vector 40 of input beam and  $K'$  vector 42 output. This scans the point in k space represented by vector 48 as discussed above. Figure 5a also shows an input k vector 60 shown dotted which corresponds to a shorter wavelength. As a concrete example, vector 40 may correspond to one line of the  $CuK\alpha$  doublet and vector 60 to the other line of the  $CuK\alpha$  doublet. The shorter incident wavelength produces a scattered beam represented by vector 62 emitted in the same direction as  $K'$  vector 42. By vector addition, this scattering probes the point in k-space given by vector 64.

The oval 54 thus represents diagrammatically the variation in the probed region of k-space caused by variation in the wavelength of the incident beam. The oval in Figure 5a is used for clarity in the Figure and is not intended to suggest an oval pattern of variation.

Vector 64 does not however probe a region of the k-space with additional sample information since the sample information is given by line 54 substantially along normal direction 14.

Thus the variation in the wavelength does not cause uncertainty in the position probed along line 52 parallel to normal 14, since the uncertainty 54 is along a different direction to the normal 14. Thus the wavelength uncertainty does not cause significant spread in the measured data.

Similarly, Figure 5b illustrates the effect of a divergent beam 66 at a different angle to nominal beam 40 yet scattering into the same direction as scattered beam 68. The uncertainty in the probed region of k-space is shown by oval 56. Again the uncertainty does not result in uncertainty along normal 52. Again it will be noted that the new radial vector 70 takes the probed point outside region 52 which contains sample information. Thus, again the moderate divergence does not cause excessive spread in the measured data.

The uncertainty 56 from beam divergence is inclined to the uncertainty 54 from wavelength variation by  $\theta$ . It should be noted that although these two sources of divergence have been described separately, the complete instrument and sample effectively operate as a single instrument with a single spread in measured data.

The third important point about the geometry used is that because the scattered vectors 42, 44 are at a small angle to the plane of the sample, i.e. at an angle close to  $90^\circ$  to the normal to the plane of the sample, the angle subtended by a finite region on the sample surface is very small at the detector. If in contrast the detector were mounted directly above the sample there would not be a one to one correspondence between the location measured along the length of the the detector and the angle  $\alpha'$  of emitted light.

Thus, the geometric arrangement used, quite unexpectedly, allows high-resolution diffractometry to take place without the use of monochromators or complex arrangements such as the double-crystal diffractometer or the multiple-crystal diffractometer. In this context, "high resolution" refers to a diffractometer that is capable of observing thickness fringes from a layer up to about 0.1 micrometer thickness.

Further, the detector 10 can record data in parallel and thus measure the scattering information corresponding to a number of scattering angles simultaneously without the need to move the sample except for very large angular scans. Hence, useable data can be collected perhaps 50 times as quickly as previously.

The inventor has realised that it is possible to select suitable sizes of source 4 and slit 6 to obtain a beam that is sufficiently non-divergent for high resolution by using the geometry proposed. Calculations have been carried out to ascertain the range of suitable values of these sizes for a preferred embodiment. The preferred embodiment is suitable for probing Si and GaAs layers using a Cu K $\alpha$  source.

The relevant parameter is the divergence at the sample 16 of the incident beam of X-rays 11 generated by the source 4 and slit 6. There is to some extent a trade-off. As the divergence is decreased below 0.06°, there is little improvement in resolution but some loss of intensity and some restriction in the range of information probed. As the divergence increases above 0.06° there is a significant increase in the wavelength band pass. For example, where the source of X-rays emits a Cu K $\alpha$  doublet the Cu K $\alpha$ 2 component starts to appear and is very clear for a divergence of about 0.25°. An angle of divergence of 0.06° has been calculated to be about optimum. Thus, in this preferred embodiment, the angular divergence of x-rays at the sample is about 0.01° to 0.20°, preferably 0.02° to 0.10°, further preferably 0.03° to 0.06°.

The x-ray source preferably has a dimension perpendicular to the beam direction in the scattering plane (i.e. the plane of Figure 6) of no more than 200 micrometers, preferably no more than 40 micrometers. In the geometric orientation proposed, the dimension out of the scattering plane can be large, even as high as 1cm. In prior art approaches, so-called Soller slits are required, a series of parallel slits only passing beams parallel to within 0.2° of the scattering plane. It is a significant benefit of the invention that these are not required.

The arrangement of the invention is not sensitive to variation in the position of the sample. A rotation of the sample about the normal 14 does not

degrade the signal, but only degrades the intensity by a factor of about 10 for a  $5^\circ$  rotation of the sample above or below optimal. Thus, it is not necessary to be excessively precise in orienting the sample - this increases the throughput of samples through the diffractometer.

5        Tilting the sample also has little effect. When the sample is tilted about an axis parallel to the surface and in the scattering plane then changing the tilt angle by  $1^\circ$  has little effect.

      The incident angle  $\alpha$  does require some adjustment to isolate the required  $\text{CuK}\alpha_1$  wavelength. The incident angle  $\alpha$  should accordingly be  
10        preferably set to an accuracy  $0.01^\circ$ , though  $0.03^\circ$  gives a workable high resolution profile.

      A variation in the sample position along the normal axis is also not critical. A motion of plus or minus 0.5mm has little effect on profile quality.

      It will be appreciated that the sample needs to be oriented to diffract  
15        from a scattering plane that is not parallel to the front plane of the sample. In particular, the optimal plane has an incident beam as close as possible to the normal to the surface and an exit beam as close as possible to being parallel to the surface. The reason for this is that the uncertainty 54 is largely along  
20        the radial direction and it is necessary that this uncertainty does not lie parallel to the sample information which lies in a region of k-space 52 parallel to the normal direction 14. However, where  $\alpha = \alpha'$  the radial vector 48 is along axis 40 and thus the high resolution disappears. Instead, the smearing and uncertainty caused by light of different wavelengths not all perfectly collimated  
25        probes a region of diffraction space that has significant scattering intensity at a number of positions, and accordingly causes smearing of the measured signal.

      Thus, whereas conventionally a 004 scattering plane may be used, the scattering of the invention should use a different scattering plane such as the 113 reflection used in the example above. The 113 is a reflection that satisfies the condition that the incident beam can be near-normal and the exit beam  
30        near parallel to the surface for the commercially important materials Si and GaAs.

In a specific implementation, the diffractometer has the following sizes. The distance of the slit to the sample  $L_1$  is 320mm, and the divergence of incident angle  $\Delta\omega$   $0.06^\circ$ . This divergence is achieved by the source and slit in parallel. The distance of the detector from the sample  $L_2$  is 240mm. Using a 113 reflection, the angles shown in the figure are given by  $2\theta$  is  $53.73^\circ$  for GaAs and  $56.12^\circ$  for Si.  $\omega$  is  $52.1^\circ$  for GaAs and  $53.3^\circ$  for Si. The detector slit width is 70 micrometers.

The inventor has calculated the resulting angular resolution  $2\theta_{\text{resolution}}$  of the instrument to be given by:

10 
$$2\theta_{\text{resolution}} \sim \{L_1 [\tan(\Delta\omega)/\sin(\omega)] \sin(2\theta - \omega)/2 + D/2\} / L_2$$

Putting in the values of the specific implementation, this gives an angular resolution of about  $0.01^\circ$  for both GaAs and Si samples. This is more than adequate for data collection. The angular resolution of  $0.01^\circ$  gives an increment corresponding to a film thickness of 1 micrometer. Therefore, the instrument is able to determine thicknesses up to about 0.3 micrometers, i.e. high resolution.

Figures 6 and 7 are measured results that illustrate the benefits of the invention. Figure 7 shows the scattering intensity of a 113 reflection measured using a twenty second static scan on apparatus according to the invention. Figure 8 shows the same sample measured using a ten minute scan on a multiple crystal X-ray diffractometer. It will be seen that results of similar quality are obtained but at a very much greater speed using the apparatus according to the invention.

Note that Figure 6 shows that the apparatus according to the invention is capable of a resolution  $2\theta_{\text{resolution}}$  of significantly better than  $0.1^\circ$ .

Not merely is the apparatus according to the invention much faster, it is also much simpler, easier to set up and accordingly cheaper.

It will be appreciated that the arrangement of the present invention cannot be achieved in a conventional high-resolution diffractometer or double-crystal diffractometer apparatus. This is because in such prior art diffractometers data from any given incident angle arrives at the detector simultaneously with data from other incident angles and accordingly it is

necessary to use an analyser such as used in the multiple-crystal diffractometer to resolve the situation.

From reading the present disclosure, other variations and modifications will be apparent to persons skilled in the art. Such variations and modifications may involve equivalent and other features which are already known in the design, manufacture and use of diffractometers and which may be used in addition to or instead of features described herein. Although claims have been formulated in this application to particular combinations of features, it should be understood that the scope of disclosure also includes any novel feature or any novel combination of features disclosed herein either explicitly or implicitly or any generalisation thereof, whether or not it mitigates any or all of the same technical problems as does the present invention. The applicants hereby give notice that new claims may be formulated to any such features and/or combinations of such features during the prosecution of the present application or of any further applications derived therefrom.

In particular, although in the embodiment described the detector is capable of detecting x-rays incident at a plurality of different incident locations in parallel, it may also be possible (though slower) to provide an arrangement that carries out the detection at a number of different incident locations in series, for example by providing a movable narrow slit in front of an X-ray detector.

## CLAIMS:

1. An X-ray apparatus for high-resolution X-ray diffraction,  
5 comprising:  
a sample stage for holding a sample having a front face with the front face oriented substantially normally to a predetermined normal direction;  
a means for generating a collimated beam of X-rays at a predetermined target location on the sample stage at an angle of between  $0^\circ$  and  $60^\circ$  to the  
10 normal direction, the beam having an angular divergence at the sample stage in the range  $0.01^\circ$  to  $0.20^\circ$ ; and  
an X-ray detector arranged laterally of the sample stage for detecting X-rays scattered by the sample to a predetermined range of angles to the normal direction, the angles in the predetermined range being in the range from  $80^\circ$  to  
15  $90^\circ$ .
2. An X-ray apparatus according to claim 1 wherein the X-ray detector has a linear resolution in the normal direction of less than 0.002 times the distance from the X-ray detector to the predetermined target location.  
20
3. An X-ray apparatus according to claim 1 or 2 wherein the means for generating a collimated beam of X-rays is an X-ray source and a slit between the X-ray source and the sample stage
- 25 4. An X-ray apparatus according to claim 3, wherein the X-ray source has a dimension of no more than 0.2mm in the direction normal to the beam in the plane containing the normal, the incident beam and the scattered X-rays.
- 30 5. An X-ray apparatus according to any preceding claim wherein the X-ray detector is an elongate X-ray detector extending in a direction parallel to the normal direction for detecting in parallel X-rays diffracted by the

sample as a function of distance along the normal direction and hence over a predetermined range of angles to the normal direction.

6. An X-ray apparatus according to any preceding claim wherein  
5 the position sensitive X-ray detector is a solid state detector.

7. Use of an X-ray apparatus according to any preceding claim to  
measure a sample having a substantially single crystal thin layer at its front  
face, the sample being mounted on the sample stage and oriented to diffract  
10 the collimated X-ray beam onto the position sensitive X-ray detector.

8. A use according to claim 7 wherein the substantially single  
crystal thin layer is a semiconductor layer.

15 9. An X-ray apparatus for high-resolution X-ray diffraction of a  
sample having a substantially single crystal thin layer, comprising:

a sample stage for holding a sample having a front face with the front  
face oriented substantially normally to a predetermined normal direction;

an X-ray source for directing X-rays towards the sample stage;

20 a slit arranged between the X-ray source and the sample stage, the X-  
ray source and slit being dimensioned and arranged to direct a collimated  
beam of X-rays at a predetermined target location on the sample stage at an  
angle of between  $0^\circ$  and  $60^\circ$  to the normal direction;

an elongate X-ray detector arranged laterally of the sample stage for  
25 detecting X-rays diffracted by the sample as a function of distance along the  
length of the X-ray detector and hence over a predetermined range of angles  
greater than  $80^\circ$  to the normal direction.

10. A method of high-resolution X-ray diffraction; comprising:  
30 providing a sample stage and an X-ray detector located laterally of the  
sample stage;

mounting a sample having a substantially single crystal thin layer material extending in a plane on the sample stage;

directing an incident collimated beam of x-rays onto the sample at an angle of  $0^\circ$  to  $60^\circ$  to the normal to the plane; and

5 measuring with the X-ray detector the X-rays diffracted by the sample to a range of angles in the range  $80^\circ$  to  $90^\circ$  to the normal to the plane.

11. A method according to any claim 11 wherein the incident beam has an angular divergence in the range  $0.01^\circ$  to  $0.20^\circ$ .

10

12. A method according to claim 11 or 12 wherein the incident beam of X-rays is in a direction from  $0^\circ$  to  $40^\circ$  to the normal to the plane.

13. A method according to any of claims 10 to 12 wherein the step of  
15 measuring the X-rays diffracted by the sample includes recording the intensity of X-rays incident on the detector simultaneously at a number of locations along the length of the detector.

## ABSTRACT

## X-RAY DIFFRACTION APPARATUS AND METHOD

A high resolution X-ray diffraction apparatus includes a source 4 of X-  
5 rays and a slit 6 to direct a collimated beam of X-rays 11 onto a sample 16 on  
a sample stage 8. Detector 10 records the intensity of diffracted X-rays as a  
function of position along its length. The geometry allows high resolution  
without the use of a monochromator.

10

[Figure 3]

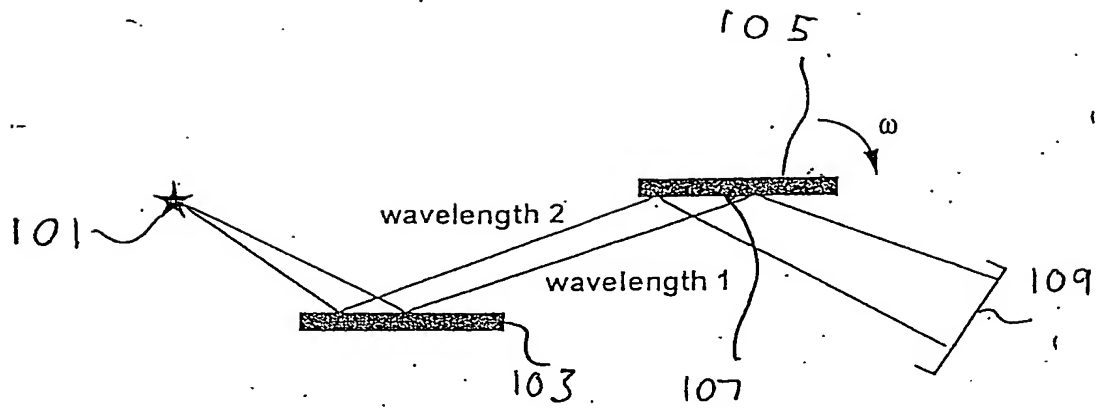


Fig 1

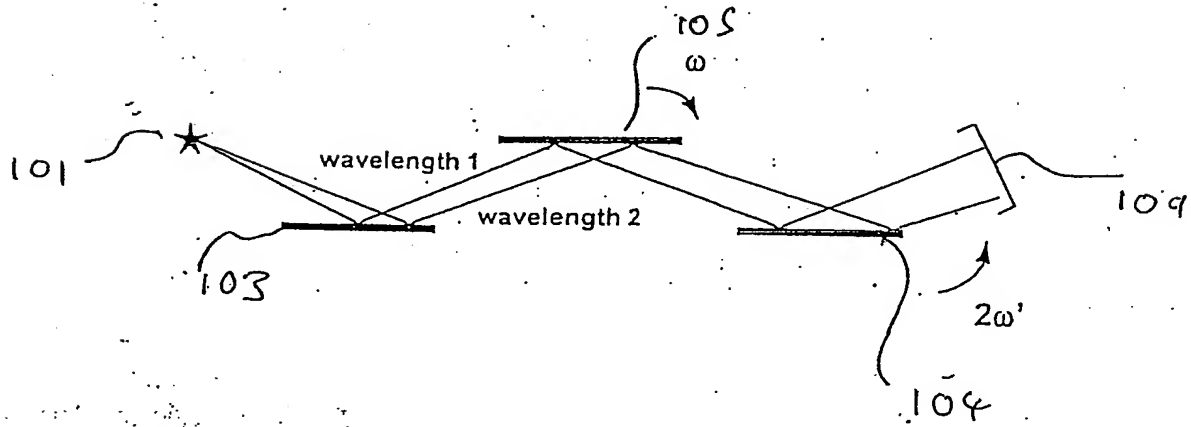
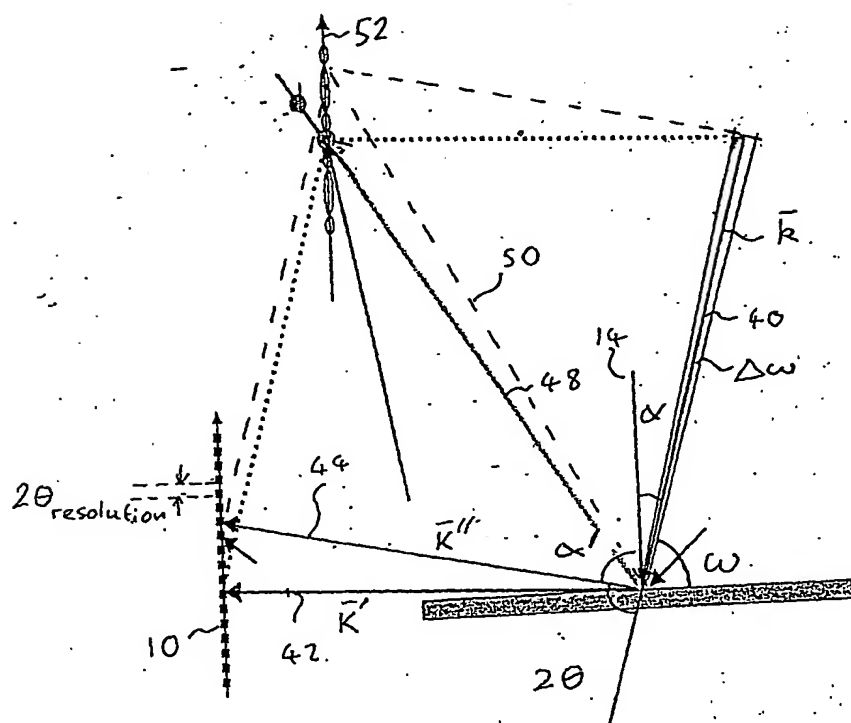
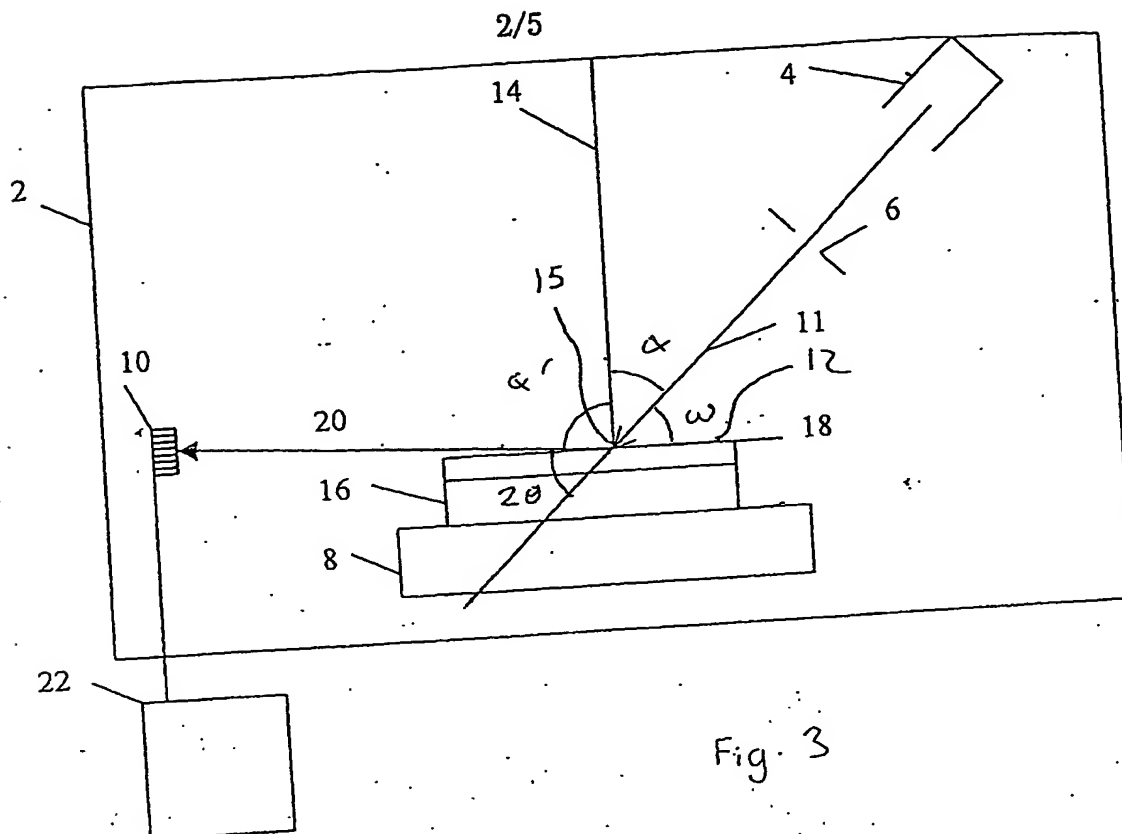


Fig 2



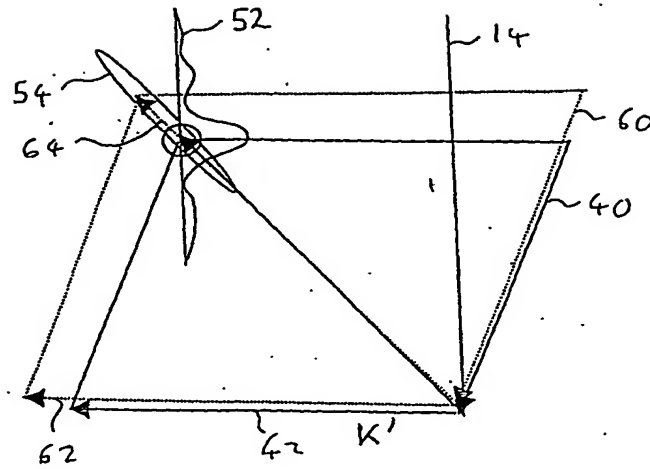


Fig. 5a

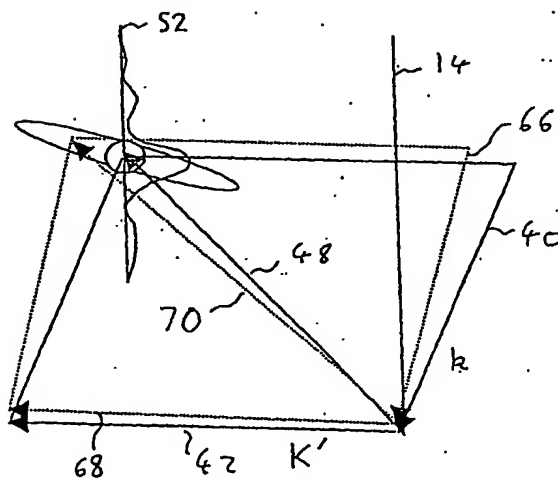


Fig. 5b

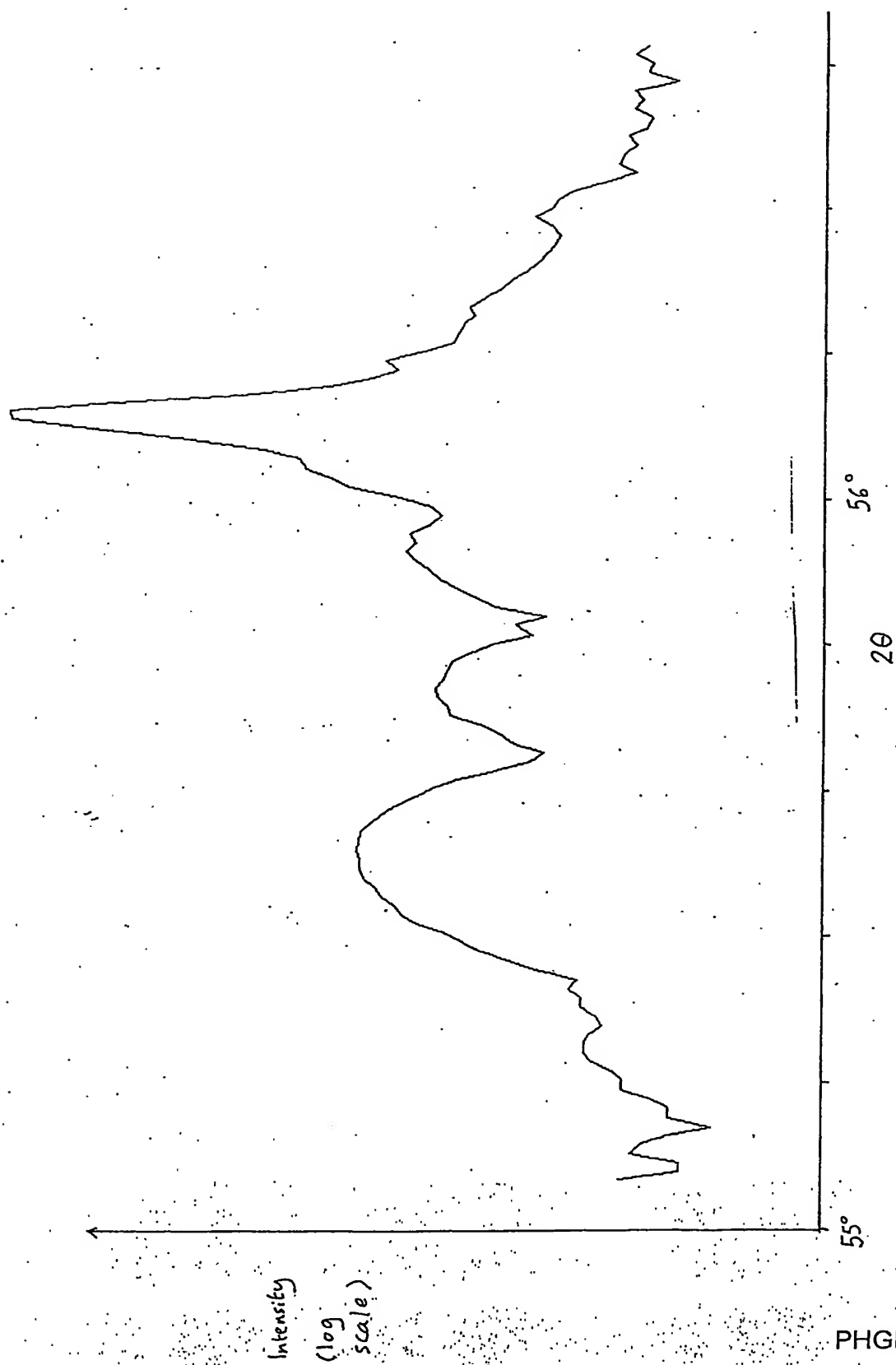


Fig. 6

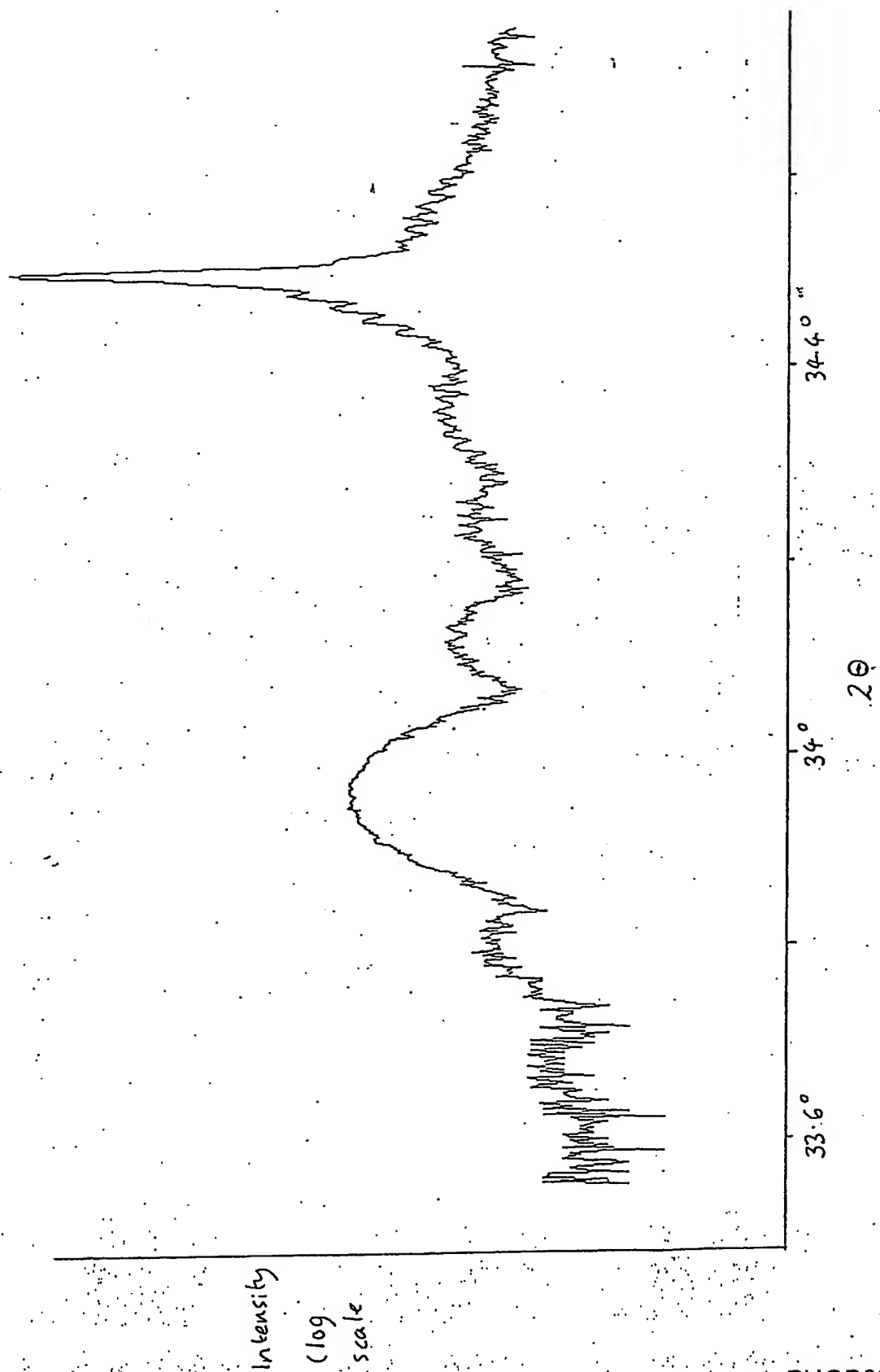


Fig. 7

**This Page is Inserted by IFW Indexing and Scanning  
Operations and is not part of the Official Record**

**BEST AVAILABLE IMAGES**

Defective images within this document are accurate representations of the original documents submitted by the applicant:

Defects in the images include but are not limited to the items checked:

- ☒ **BLACK BORDERS**
- ☒ **IMAGE CUT OFF AT TOP, BOTTOM OR SIDES**
- ☒ **FADED TEXT OR DRAWING**
- ☐ **BLURRED OR ILLEGIBLE TEXT OR DRAWING**
- ☒ **SKEWED/SLANTED IMAGES**
- ☒ **COLOR OR BLACK AND WHITE PHOTOGRAPHS**
- ☒ **GRAY SCALE DOCUMENTS**
- ☐ **LINES OR MARKS ON ORIGINAL DOCUMENT**
- ☐ **REFERENCE(S) OR EXHIBIT(S) SUBMITTED ARE POOR QUALITY**
- ☐ **OTHER:** \_\_\_\_\_

**IMAGES ARE BEST AVAILABLE COPY.**

**As rescanning these documents will not correct the image problems checked, please do not report these problems to the IFW Image Problem Mailbox.**